

High Speed Spectrum Imaging of Raney Nickel Alloy Using a Large Area Silicon Multi-Cathode Detector (Vortex-EMTM) and Event Streaming Technique

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This paper reports on our continuous efforts to improve elemental mapping in electron beam microanalysis by taking advantage of the new Vortex-EMTM detector [1]. The detector was integrated into a JEOL 840 SEM at the National Institute of Standards and Technology (NIST) and then coupled to the innovative 4pi RevolutionEDX system [2]. Raney Nickel Alloy, a well-known chemical catalyst rich in microstructures, was chosen for high speed spectrum imaging.

The Vortex-EMTM incorporates a silicon multi-cathode detector, a type of drift detector. At a peaking time of $\sim 6 \mu\text{s}$, an energy resolution of 125 eV FWHM at 5.9 keV is achievable. An output rate as high as 600 kcps is obtained at a processor peaking time of 0.25 μs , with virtually no change in peak position, nor degradation in energy resolution. The detector has excellent sensitivity to low energy x-rays, allowing for detection of low atomic number elements down to boron. The detector has proven resistant to radiation damage by high energy electrons in TEM applications, which has plagued many other types of x-ray detectors. The detector operates with thermoelectric cooling and passive heat transfer to the ambient without using any moving parts.

The 4pi RevolutionEDX system uses a new Event Streaming technique to collect x-ray spectrum images at electron imaging speeds with zero overhead [3]. With this technique, a full spectrum can be collected for each pixel with a very short dwell time (in microseconds). Event Streaming treats x-ray events as an alternative signal source, outputted directly from the x-ray pulse processor's auxiliary interface bus into the scan generator. The events are then interpreted and combined with the x-ray position information into pixel events. The pixel events are packetized and streamed to a host computer where they are buffered and stored. The whole process takes place at the hardware level with an extremely high speed and is thus highly efficient. Since host processing is not linked to data acquisition, a high degree of live interactive analysis is possible, including real-time analysis of conditions, which eliminates the need to restart the acquisition. The analysis routines simply replay the saved events, eventually catching up to the live events.

Spectrum image maps were acquired under several different conditions in the JEOL microscope at NIST. With the high output rate of the Vortex-EMTM detector, fewer frames were required to achieve a high quality composition map, compared with conventional mapping techniques. In conjunction with the high speed processing of the 4pi system, the mapping time was significantly shortened to only a few minutes, a process which normally requires hours with a Si(Li) detector and conventional processing electronics. A sample composition map acquired using the system

described, is shown in Figure 1. Additional spectrum image maps and spectra will be presented and discussed.

1. S. Barkan, V. Saveliev, J. Iwanczyk, L. Feng, C. Tull, B. Patt, D. Newbury, J. Small, N. Zaluzec, "A New Improved Silicon Multi-Cathode Detector (SMCD) for Microanalysis and X-Ray Mapping Applications", *Microscopy Today*, November 2004.
2. RevolutionEDX EDX Analysis and Digital Imaging System, 4pi Analysis, Inc., Durham, NC USA (www.4pi.com).
3. Scott D. Davilla, "Event Streamed Spectrum Imaging (ESSI) – Concurrent EDX and Its Application to Particle Analysis", to be published in *Scanning 2006 Proceedings*.

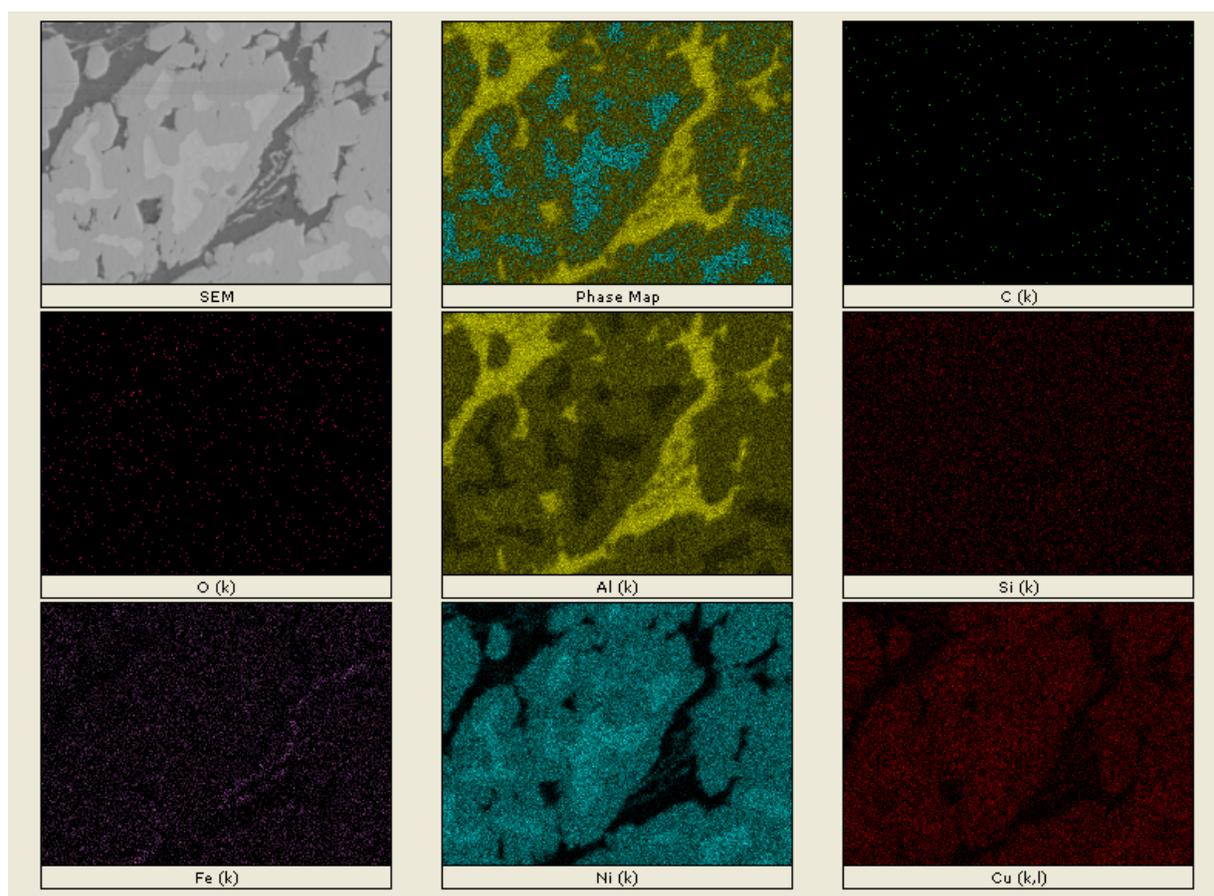


Fig. 1. Phase and elemental maps of a Raney Nickel Alloy sample. From left to right, *upper row*- SEM, Phase Map, C(K); *middle row*- O(K), Al(K), Si(K); *bottom row*- Fe(K), Ni(K), Cu(K,L). Size: 640 x 480; peaking time: 1 μ s; acceleration voltage: 20 kV; output count rate: 100 kcps; dwell time: 32 μ s; number of frames: 20; total mapping time: 196 sec.